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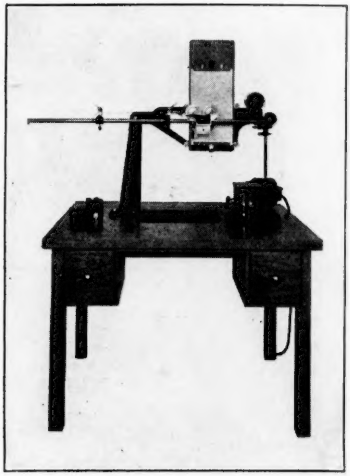

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TEXTILE RESEARCH

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An Instrument for the Study of Compressional Creep and Creep Recovery of Yarns and Fabrics

By K. R. FOX, S.M.,* and E. R. SCHWARZ, F.T.I.†

A Textile Foundation Publication

Abstract

A brief outline of previous developments in compression testing of textiles is given and the details of construction of an instrument intended for the study of the compressional creep and creep recovery of yarns and fabrics are noted. Preliminary results are presented to show the characteristics of the instrument. Later papers will discuss specific tests.

Introduction

TEST methods involving compression are being given increasing attention by the textile technologist. Any determination which involves pressure of one unit against another involves compression; thus, thickness measurements of yarn or fabric, when carried out by means of the usual thickness gage having a presser foot and anvil, are compression tests. Measurements of the compressibility of mattress stuffing materials, carpets, or insulating pads are additional obvious examples.

In addition to the compressibility, the recovery from compression is of considerable interest. This recovery is known as compressional resilience and may be elastic in nature if the deformation resulting from the compression is instantly and completely recovered. It may be in the nature of creep if the recovery takes place only relatively slowly. There may also be (possibly temporary) non-recoverable compression which is measurable and often of interest.‡

Because of the fact that textile materials in general exhibit the properties of plastic substances and are therefore very considerably affected by the time factor in tests which involve change of shape with load application it is necessary in testing to consider a definite schedule of load application

* Research Assistant for the Textile Foundation at M. I. T.
† Professor of Textile Technology, M. I. T.

‡ While this paper is concerned entirely with compressional resilience, it should be noted that recovery from bending, from tensile strain, or from torsional strain is also possible of consideration as other forms of resilience.

in order to determine the effect of the rate of loading and the effect of cyclic loading upon the results. It is further necessary to evaluate the resilience in terms of work done or in terms of energy rather than in terms of simple deformation. It will be easily understood that a specimen may be deformed from one size to another with the expenditure only of considerable energy or conversely with very little energy. The recovery after the deformation may require a substantial amount of work which has been stored up as potential energy in the sample when the deformation originally occurred and which can be restored in whole or in part as the material comes back more nearly to its original condition. If the results of a compression and recovery test are plotted it will be seen that between the initial and final points on the curve any number of lines may be drawn, each one indicating a certain load-deformation behavior. The area beneath any one of these lines is a measure of the energy involved and will be different for each path taken. It is not enough therefore simply to measure the deformation produced at any particular load and to report the resilience in terms of this deformation. It is of importance to know how easily the recovery was brought about or with what difficulty the compression was produced.

Apparatus Available

In general three types of apparatus have been devised for handling compressional resilience testing. Several of these involve the testing of fiber masses and here some container is essential to keep the fiber mass under control. Usually one of cylindrical shape is employed. Such an apparatus was devised by the United States Department of Agriculture; another was designed, built and calibrated by Peterson and Wenberg for the Massachusetts Institute of Technology as a thesis in 1929.* In use, loads are applied at convenient increments and the test is slow.

A dynamic test of similar nature is reported by D. S. Robinson.† In this test a series of plant fiber masses were repeatedly compressed in a cylinder by means of a piston which could be raised and lowered rapidly by means of a motor-driven disk which acted in the manner of a cam against a lever connected to the piston by means of a chain. Graphic record was kept of the second, third, tenth, twentieth, fiftieth, one hundredth and one hundred fiftieth compressions. Preliminary tests indicated that the compressions should not be more than two per minute and it was found that it required about seven to nine seconds for the fiber to rise because of its own resilience. As the plunger was lifted the compressed fiber mass rose a certain amount nearly instantaneously and the remainder of the rise was gradual after the plunger was off the fiber and took perhaps four to five seconds. The manner of behavior will obviously vary with the type of fiber which is used.

The apparatus of Wenberg and Peterson was improved by Dockstader ‡ using the principle of the inclined plane (constant rate of load) which was developed by the H. L. Scott Company for use in tensile testing machines and is patented by them. The instrument was built under special arrangement

* This machine is illustrated on page 356 of the second edition of the Wellington Sears Handbook of Industrial Fabrics.

† *Textile Research*, Vol. 8, July 1938, p. 310, 316.

‡ M. I. T. Master of Science Thesis, 1935, E. K. Dockstader. "Design of an Automatic Recording Power Driven Machine for Testing the Resilience of Textile Fibres."

with the H. L. Scott Company and provides an autographic record in multiple of as many complete compression-resilience cycles as may be desired. The apparatus is of the cylinder and plunger type but the load is applied at a constant rate throughout the compression and recovery portions of the cycle and operates with a minimum of attention. From the resulting charts it is possible to measure the areas representative of the energy involved directly. Reference to Figure 1 will show diagramatically how the

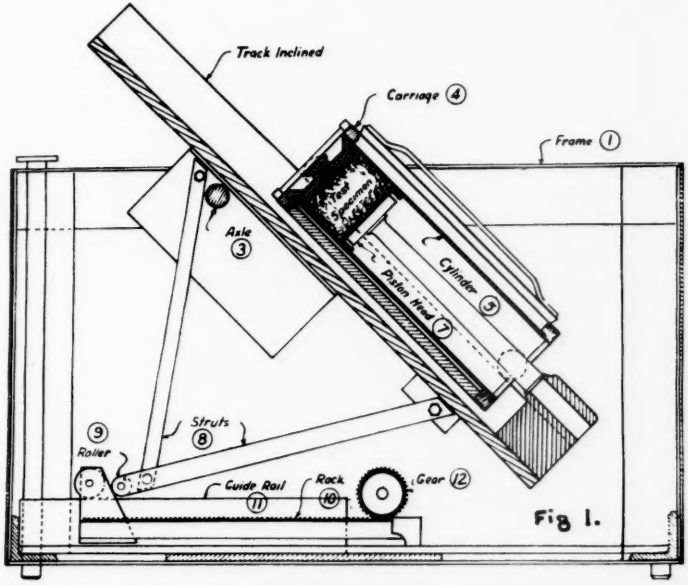

Fig 1.

machine is constructed. The apparatus has been used on samples of bulk fiber and also on cut specimens of carpet or other fabrics in multiple.

The principle of the piston and cylinder has been modified somewhat by Saxl * who uses a simple triple beam balance to support a beaker on the platform. An accessory vertical support carries a rack and pinion which controls the movement of a plunger, the descent of which into the beaker is measured by means of graduations on the vertical support. If the plunger is lowered into a beaker containing a weighed sample of fibers the balance can be brought to equilibrium and the weight necessary to do this can be recorded and plotted against the depression of the plunger into the beaker. The loading rate in this instrument obviously is not continuous and it is necessary to control the relaxation time carefully between successive loadings and either to apply the load in successive equal steps with a known relaxation period and bring the balance into the equilibrium posi-

* *Rayon Textile Monthly*, February 1938.

tion by adjusting the plunger or to adopt the alternative method of equal increments of plunger travel and a variable loading. The application of the load is not under particularly good control as compared with the constant load rate machine of the inclined plane principle or as compared with the chainomatic loading device to be described in the following paragraph.

All of the above instruments employing the principle of cylinder and plunger are to be criticized due to the fact that there is a friction effect between the fiber mass and the walls of the cylinder and in certain cases this may also apply to the friction of the fiber against the sides of the plunger. This effect, however, is believed to be relatively unimportant in comparison with the magnitude of the forces used in the compression and recovery of the sample.

For the study of yarns and fabrics in compression and particularly for their recovery from compression Schiefer * devised an instrument at the National Bureau of Standards which is known as the Compressometer and which indicates the degree of compression on a specimen by means of a standard thickness gage. Either a calibrated spring or other suitable device is used to produce the pressure of the foot of the instrument against the specimen and the loading is intermittent and with a relaxation period recommended as fifteen seconds. The instrument is not adapted in its present form to the measurement of bulk samples of fiber but rather for flat pieces of fabric or yarns wound on a card or placed against a known type of surface. It has also been used for evaluating the crush resistance of fabrics which have been subjected to special finishing treatments.† It was found that on a pile fabric, decrease of thickness was rather gradual until the pile collapsed. When the pressure was released, complete recovery to the original thickness was not obtained. Similar tests have been conductd on the instrument designed and built at M. I. T. and to be described in the following paragraphs and these results are confirmed and will be amplified in more detail in a subsequent paper.

Still another method for making measurements of compression effects and compressional resilience was developed by C. G. Winson ‡ in which a balloon is used to confine the sample and where the pressure is applied in all three dimensions rather than in one direction as in previously described pieces of equipment. The balloon is filled with a weighed sample of fiber and is attached to the end of a tube, the whole being enclosed in a glass jar. The balloon and the glass jar are then evacuated equally and their pressures read from monometers. The rubber balloon has a definite volumetric displacement which is determined in advance and upon readmission of air to the jar and the establishment of an equilibrium condition the pressures can be redetermined on the resilience cycle. The tests are repeated until the difference between the pressure within and without the balloon is, say, fifty centimeters. Air is then evacuated from the jar in steps until the pressures are again equalized and the pressures determined at each step. The volume of the internal system at one condition is proportional to the volume of the internal system at any other pressure. The difference be-

* *Bureau of Standards Journal of Research*, Vol. 10, June 1933, pp. 705–713; *Textile Research*, Vol. 3, October 1933, pp. 505–513. "The compressometer an instrument for evaluating the thickness, compressibility and compressional resilience of textiles and other materials."

† H. F. Schiefer. *American Dyestuff Reporter*, Vol. 26, 1937, p. 667–9.

‡ *Journal Textile Institute*, Vol. 27, 1932, p. 386–393. "Report on the methods of measuring resilience of wool."

tween the pressure within and without the balloon plotted as abscissa against the ratio of the volume of the balloon at various pressures to the volume at a standard pressure gives a curve indicating the cubic compression of the wool against pressure. Definite hysteresis is found between the unloading and loading curves so that the findings should be given in terms of energy and were so reported by the author.

In order to obtain a considerable degree of precision and a wide range of control for the application of the load, a modified instrument has been designed and built in the Textile Laboratory of the Massachusetts Institute of Technology. It was primarily designed to obtain general information in the field of compression and compressional resilience of a range of fabrics differing in structure or fiber composition or both and including pile fabrics of various characters. It is also to be used intensively for a further study of the superposition principle * which is being investigated for the Textile Foundation for textile materials in tension as a part of the same program. For this work it is necessary to have an extremely smooth load application, extreme sensitivity in the recording of deformation and, further, as great a degree of elimination of friction as possible. The device is able to carry through a series of cyclic loadings under almost any desired condition and is also suited to the recording of behavior of a sample under a constantly applied load or one which is applied for a known period, released for a known period and reapplied in the usual fashion for the determination of creep and creep recovery properties.

Apparatus

The apparatus for the present resilience tester may be subdivided into three main mechanisms. The first of these can be designated as a calibrated plunger (see Fig. 2A). This consists of support made up of a half inch steel plate mounted on three leveling screws. These screws have knurled heads and can be used to level the plate. Equidistant from these three supporting screws is mounted the plunger itself. This consists of a steel shaft which has been cut into a precisely fitted screw with 40 threads per inch producing a lead of 0.025 inch. The top of this plunger has been equipped with a graduated dial of 250 divisions. One division on this dial will then represent a plunger travel of 0.0001 inch. Attached to the other end of this calibrated plunger is an interchangeable presser foot with an area of either 1 or 2 square inches. It is necessary that the foot in use either advance or retreat vertically and yet not turn about its own axis. To accomplish this, the contact between the plunger and the foot was constructed on the cup and ball principle. It is then possible to advance the foot and the friction of the point contact will not be sufficient under load to turn the foot about its axis. Each foot is provided also with an annular slot into which a set screw from the plunger may be advanced in order to prevent the foot from becoming disengaged from the plunger when not being used.

The second part of the apparatus is the duralumin weighting lever (see Fig. 2B) provided with two knife edges, a table for the sample being tested, and a counterbalancing weight. One knife edge is inverted and is used for transmitting the load to the lever, while the second knife edge

* "Textile Materials and the Time Factor," H. Leaderman. *Textile Research*, Vol. 11, Feb. 1941, p. 171–193.

provides the fulerum for the lever. The entire lever is supported on this knife edge and pivots in a pair of 90 degree agate bearings. The table for the sample has two engraved circles cut on its surface possessing areas corresponding to the presser feet on the end of the plunger. The center of these circles has been so placed that the lever ratio calculated to the circle center is 4. A chainomatic * system of loading is employed on the weighting lever and the lever ratio increases the dead weight of chain four times. The counterbalancing weight can be used to balance the weight of the sample after it has been placed on the table for testing.

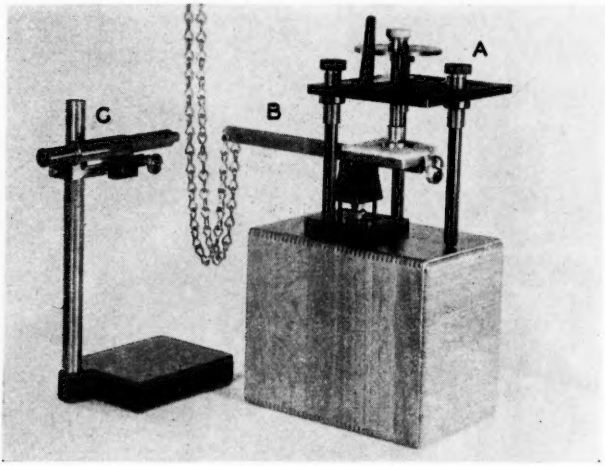

FIG. 2.

The third piece of mechanism is a low power telescope (see Fig. 2C) provided with a cross hair in the ocular. This viewing device can be focussed on a fine line on the side of the lever in order to establish a zero reading for all observations.

Testing Method

Before any tests can be run, it is necessary to insure that the apparatus is level. The steel plate carrying the plunger must first be levelled by observing the circular levelling bulb on the surface of the plate. The weighting lever should now be placed under the plunger so that the presser foot exactly covers its corresponding engraved circle on the lever table. A portable levelling gauge may now be placed on the lever and the plunger

* This method of loading provides different lengths of chain hanging on the lever knife edge. The free end of the chain can be lowered or raised by the use of a suitable arrangement of cords and pulleys in order that the load can be increased or decreased at will. The chain is composed of 78 links with an average of 0.424 ounce per link.

position may be altered until the lever is exactly level. The reading on the graduated dial at this position indicates the zero setting of the plunger. The telescope should now be focussed on the engraved line on the side of the lever. When this has been done, the apparatus is in a condition to be used for testing.

Before any sample is inserted beneath the plunger, the weighting chain should be adjusted so that the minimum load to be used is acting on the lever. In the tests mentioned here, three links were taken as the zero load. The chain should now be disconnected by slipping the links from the knife edge. The sample may then be placed on the lever table and its weight compensated by the counterbalancing weight. When this is done, the chain may once again be hung over the knife edge taking care not to subject the sample to shock when it is brought in contact with the plunger. The plunger height should now be adjusted until the image of the engraved line on the lever coincides with the cross hairs in the telescope. The difference between the dial readings when the lever is in balance and the zero setting represents the sample thickness with the arbitrary zero load. The pressure on the sample may then be increased by adding to the length of chain acting by gravity on the lever arm. The lever should be kept in balance during any change in load by turning down on the plunger to compensate for the presser foot penetration into the fabric. It is necessary to maintain equilibrium throughout the test to prevent any eccentric loading on the sample due to the angularity of the lever if it were allowed to swing from its level position. The differences in the plunger settings represent penetrations of the pressure foot into the sample and these readings can be correlated with their respective loading values to give data for suitable plotting of load versus penetration. The cycle can be reversed when the maximum load has been reached so that the recovery of the sample can be depicted at the successively decreasing loads. By plotting the values of load versus penetration and recovery, the resilience of the cycle can be calculated.

It should be noted that the time elapsed between loading applications or removals is a very important factor in depicting the resilient properties of any samples. In addition, the rate of loading from one setting to another is of utmost importance and should also be stated in reporting any decisive quantitative results. Further experiments on these as well as other variables are now being conducted and will be reported in a later paper.

Calculation of Resilience

Many methods for calculating resilience have been offered by various authors but the one adopted in this paper utilizes the conception of energy. In mechanics, the term resilience is used to denote the *amount of work* which a body under strain is capable of doing in returning to its unstrained state; that is, *the resilience is the potential energy due to strain.* This value should not be confused with the energy expended in deforming the sample as they are alike only in the case of a true elastic body, that is, one that does not exhibit a strain lag or "hysteresis" upon the removal of stress. As test samples are not all of the same construction, a more representative value is used, called *per cent resilience.* This value is the amount of energy returned by the sample upon the removal of load expressed as a per cent of the energy expended by the testing machine in deforming the sample.

Consider, now, the typical curves as presented in Fig. 3. The loading phase of the cycle is represented by the curve OA indicating the penetration of the plunger with an increase in load. The removal of load is depicted by the curve AB. It follows that the energy expended in deforming the sample can be calculated from the area OAC under the loading

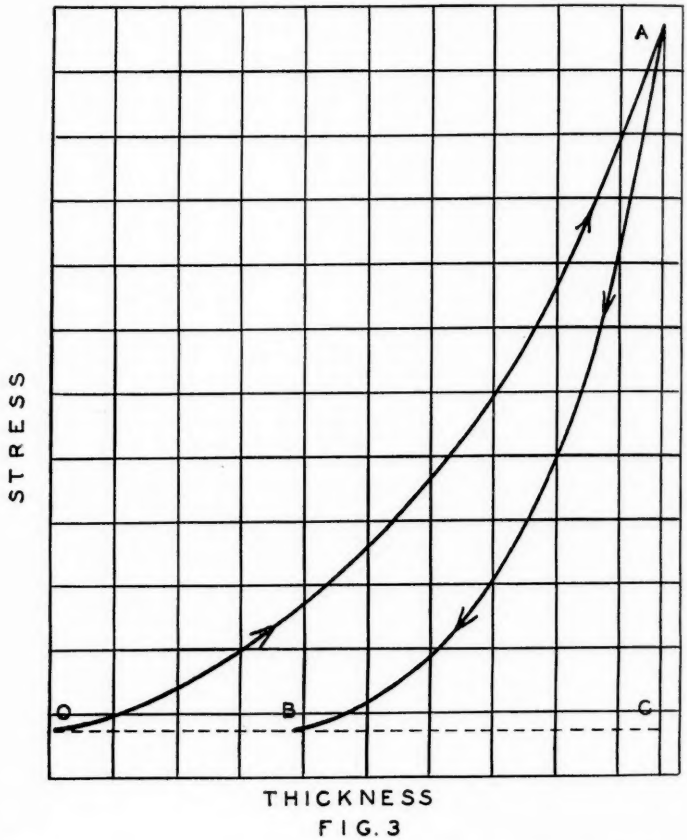

FIG. 3

curve. The energy returned to the testing machine is represented by the area ABC under the recovery curve. The difference between these areas or OAB therefore represents the energy lost or the energy that is unrecoverable. The ratio of the area ABC to the area OAC expressed in per cent will give the per cent resilience. If the curve AB were superimposed on the curve OA, the sample would possess 100 per cent resilience or, if no

work were recovered, the recovery curve would be AC and thus represent a resilience of zero per cent. The resilience of a sample may also be represented by the lost energy area OAB when expressed as a per cent of the area OAC. In this case, a sample possessing complete and instantaneous recovery would be represented by a zero resilience whereas a 100 per cent resilience would mean no recovery of work whatever. This latter method of calculating resilience is not so desirable as the first because of the confusion involved in an inverse relationship.

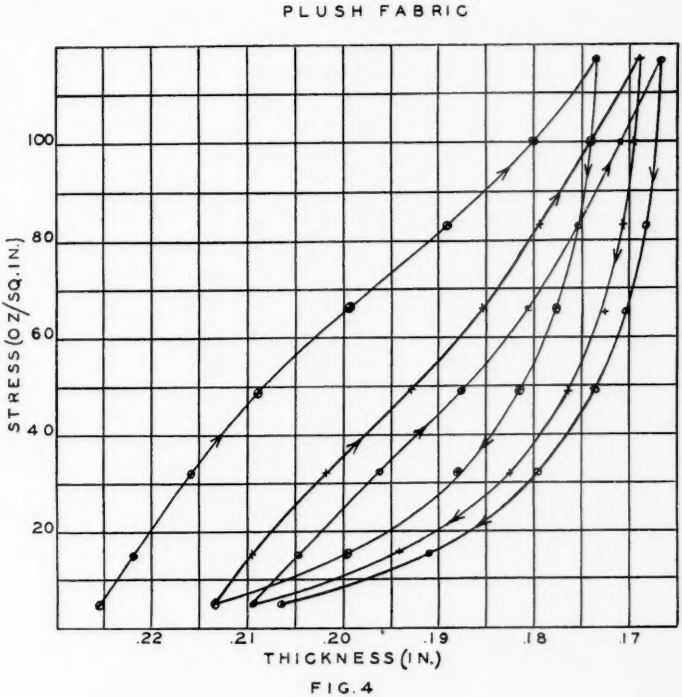

FIG. 4

The easiest and most rapid method for calculating the areas under the curves is by means of a planimeter. This is an instrument which mechanically obtains the area of any figure by simply traversing the perimeter of the area being measured. The area of the figure can then be read from a recording drum on the instrument. A second method which may be employed to advantage if a planimeter is not available is the "counting squares method." If the curves are plotted on cross section paper, the number of squares under each curve is proportional to the energy in question and may be used for calculations. This method may be somewhat

laborious at times but the results obtained are excellent. A third method may be described as the "cutting out method." This method is based on the assumption that paper on which the plot is made has a uniform weight per unit area. The area under the loading curve should be cut out from the sheet and weighed on a sensitive balance. The area under the recovery curve can be cut away from this paper sample and also weighed. The ratio of these weights expressed as a per cent will then be the resilience.

A few of the typical curves that can be obtained by the use of this instrument are included in Figs. 4 to 7. Fig. 4 presents the type of diagram resulting from successive loading and recovery cycles on a plush fabric. It is obvious that successive cycles are of a different shape indicating that the loading history is necessary before any quantitative results can be reported. This type of test involving repeated cycles approximates the kind of treatment experienced by a plush under repeated compression.

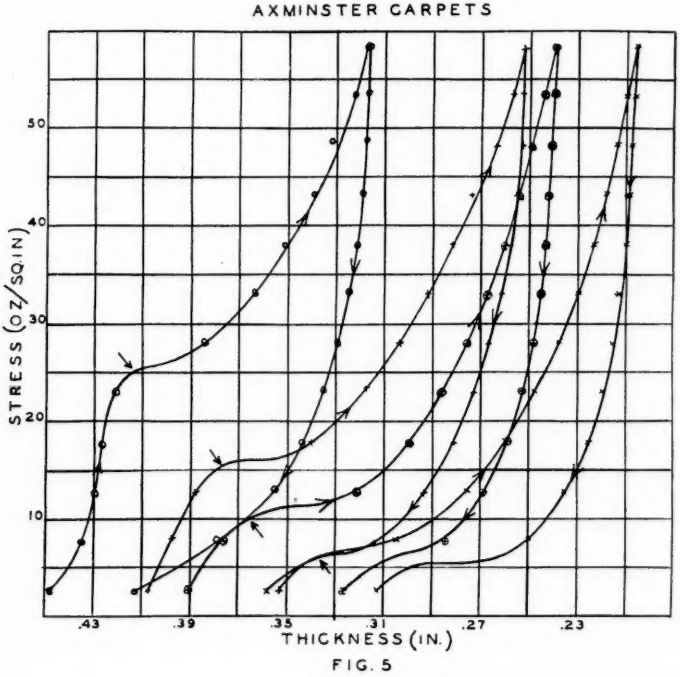

FIG. 5

Fig. 5 presents the first cycle of loading and recovery for four different Axminster rugs of varying shot settings. It is quite obvious from the shape of the curves that the carpets exhibit remarkably different compressional characteristics. In addition to the per cent resilience that can be

calculated from these curves, there is another characteristic of each curve that clearly indicates the rigidity of the pile. The sections indicated by the arrows may be called the zone representing the "pile rigidity" of the carpets. It is noted that this zone shifts for the four carpets of fewer shots per inch. This zone may very well be considered indicative of the quality of the rug being tested.

Fig. 6 presents the data obtained from testing two samples of Congo cloth one of which had been treated with a crease-proof resin. The results

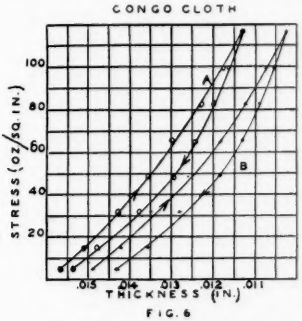

FIG. 6

from these tests indicate the treated fabric (A) possesses a resilience of 81.2% as contrasted with the resilience of the untreated sample (B) of 75.0%.

Results can also be obtained on testing single layers of yarns as shown in Fig. 7. This suggests a field of investigation correlating the twist or

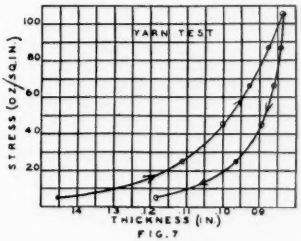

FIG. 7

stock in the yarn with the desired resilient properties in the composite fabric. Research along this line is now being conducted and will be reported at a later date.

Base-Combining Capacity of Cotton

By ARNOLD M. SOOKNE and MILTON HARRIS *

A Textile Foundation Publication

Abstract

It is well recognized that cellulosic materials contain acidic groups which bind ions and accordingly influence the electrical conductivity, the dyeing, and the finishing of the fibers or fabrics.

In an earlier investigation, the relationship between the ash content of cotton fibers and their acid-binding capacity was developed. To study further the acidic properties of cotton, the investigation has now been extended to include the direct estimation of the acidic groups in the fiber by titration with alkali. Both monovalent and divalent cations were used in this work.

When dewaxed cotton is freed of its cationic ash electrodialytically, it binds a maximum of 0.065 milliequivalent per g. of either silver or calcium ions, while depectinized cotton binds only 0.010 milliequivalent per g. These values confirm previous estimates of the carboxyl contents of these samples obtained by titration with acid. The identity of the base-binding capacities when mono- and divalent ions are used is a strong indication that the binding of these ions results from an acid-base reaction rather than from some undefined adsorption process. This conclusion was substantiated by esterifying the acid groups of eelctrodialyzed depectinized cotton with diazomethane, which thus reduced the base-binding capacity to nearly zero.

It is shown that the maximum silver-binding capacity can be obtained only by the use of the silver salt of a very weak acid. When the salt of a strong acid is used a sufficient number of hydrogen ions are in the solution to compete with the silver ions for the acidic groups of the fiber.

The silver-binding capacities of electrodialyzed dewaxed and electrodialyzed depectinized cotton are greater than those of dewaxed and depectinized samples, respectively. This result is explained in terms of competition of silver ions with the cations already on the acidic groups of the fiber.

The number of acidic groups in depectinized cotton is not altered by progressive treatment of the fiber with alkali, a fact which lends support to the tentative conclusion that the acidic groups in depectinized cotton are an integral part of the cellulose molecule.

* Representing the Textile Foundation Research Associateship at the National Bureau of Standards.

I. Introduction

COTTON and other naturally occurring cellulosic materials bind acid by an exchange process in which the hydrogen ions of the acid replace the cations associated with acidic groups of the fiber.[1, 2] By application of this concept of ion-exchange to an investigation of the acidic properties of cotton it has been possible to allocate tentatively the acidic groups to the principal constituents of the fiber.[2] Thus, in mature cotton, most of these groups are part of the pectic substance; those not in the pectic substance are assumed to be part of the cellulose molecule. Considerable interest is attached to the latter assumption, especially since, if true, it might shed some light on the complex cellulose molecule.

In order to study further the acidic properties of cotton, our earlier work has now been extended to include the direct estimation of the acidic groups in the fiber by titration with base, using both monovalent and divalent cations.

II. Experimental Procedure

1. MATERIALS

All of the measurements were made on cotton from the same batch used in the earlier work.[2] The preparation of samples of "dewaxed cotton" by extracting raw cotton with alcohol and "depectinized cotton" by extracting dewaxed cotton with alkali has been described.[2]

Some samples of dewaxed and of depectinized cotton were freed of their cationic ash by an electrodialytic procedure as follows. About 75 g. of cotton was wetted out with a 0.01 *M* solution of hydrochloric acid at room temperature, and then electrodialyzed in a large cell until the conductance of the liquid in contact with the fiber approached that of distilled water. Quantitative determinations showed that the cationic ash of these samples was less than 0.004 M–eq/g. (milliequivalent per gram). These samples will henceforth be referred to as "electrodialyzed dewaxed," or "electrodialyzed depectinized cotton."

All samples were conditioned at 21° C. and 65-per cent relative humidity before weighing. The moisture contents of the fibers were determined by drying representative samples at 105° C. for 2 hours in a vacuum oven.

2. METHODS

Determination of the silver-binding capacity.—Samples of cotton weighing about 5 g. and containing a known amount of moisture were immersed in 100-ml. portions of solutions of silver salts at 25° C. The amount of silver bound by the sample was determined by titration of 50-ml. aliquots of the original solution, and of the solution in equilibrium with the sample, using the Volhard method.[3] Preliminary measurements at several concentrations of silver ion indicated that equilibrium is attained in about 24 hours at 25° C.

The solution of silver *o*-nitrophenolate used in some of the measurements was prepared by adding an excess of silver oxide to a solution of *o*-nitrophenol at 70° C., and then filtering off the undissolved material. The silver *o*-nitrophenolate solutions were therefore saturated with respect to silver oxide.

Determination of calcium-binding capacity.—The calcium binding capacity was determined by the electrodialytic method previously described.[4] In the present investigation, however, a six-unit apparatus was used,[6] and each of the determinations reported represents the mean of 4 determinations, corrected for 2 blanks.

Determination of pH.—pH values of the suspensions were measured with a McInnes and Belcher-type glass electrode and a vacuum-tube potentiometer, using a cathode-ray tube as null indicator. The pH values were referred to potassium acid phthalate, 0.05 *M*, to which was assigned a pH value of 4.01.[6]

Temperature.—The solutions of silver salts in contact with the fiber were kept at 25° ± .02° C.

III. Results and Discussion

1. COMBINATION WITH SILVER IONS

The use of silver salts for the determination of carboxyl groups in cellulosic materials has been suggested by Rath and Dolmetsch.[7] They found that only very little silver was bound by the fibers in silver nitrate solutions but that a significant amount was bound in solutions of silver acetate. The latter salt was accordingly suggested for use in this connection. As is shown below, however, not all of the carboxyl groups bind silver when the cellulosic material is immersed in a dilute solution of silver acetate.

The combination of electrodialyzed dewaxed or depectinized cotton with silver ion may be represented by the equation

$$R-COOH + AgA \rightleftharpoons R-COOAg + HA \qquad (1)$$

where —COOH represents the acidic groups of the fiber, and AgA represents the particular silver salt under consideration. The extent to which the reaction takes place is determined principally by the competition of silver with hyrogen ions for the acidic groups of the fiber. When silver nitrate is used, a strong acid (nitric) would be formed according to eq. 1 and this would result in a relatively high concentration of hydrogen ions. Under such conditions, not all of the acidic groups of the fiber would be expected to bind silver. If, however, a salt such as silver acetate is used, a weaker acid (acetic) is formed and accordingly a greater amount of silver is bound. When silver *o*-nitrophenolate is used, a still weaker acid (*o*-nitrophenol) is formed and the amount of silver bound correspondingly increases.

In dewaxed cotton, or in depectinized cotton which has been washed with calcium hydroxide, the acidic groups are already in the salt form, and the reaction of these fibers with the silver salts may therefore be represented by the following equation

$$R-COOM + AgA \rightleftharpoons R-COOAg + MA \qquad (2)$$

where M represents the cations in the cotton

Here the extent to which the reaction takes place is determined principally by competition of silver ions with these cations for the acidic groups of the fiber. This system, however, is considerably more complex than that shown in eq. 1, since M may represent a number of different cations. In addition, it must be pointed out that hydrogen ions will also be present

(because of hydrolysis of the silver salts, AgA), and these will also compete for the acidic groups of the fiber.

It is obvious that where cations are already present in the fiber or where competition of hydrogen ion (formed according to eq 1) becomes appreciable, combination with silver ions may be increased by increasing the concentration of silver salt. Unfortunately, data obtained at higher concentrations of salt are subject to an error arising from the selective sorption * of water by the fibers and for this reason, concentrations above 0.01 *M* are avoided in the present work.

The combination of electrodialyzed dewaxed and electrodialyzed depectinized cotton as a function of concentration of silver (at equilibrium) is shown graphically in figure 1. The data were obtained in silver *o*-nitro-

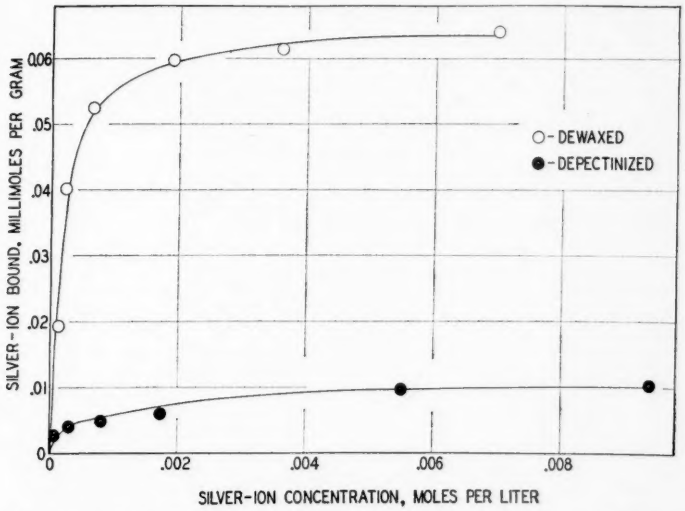

Fig. 1.—Combination of Electrodialyzed Dewaxed and of Electrodialyzed Depectinized Cotton with Silver Ion at 25° C.

phenolate solutions. The pH values of the solutions were high enough in all of these experiments so that there could be no appreciable combination of hydrogen ions by the acidic groups of the fiber. The flattening of the curves at higher concentrations indicates that the maximum silver-binding capacity has been approached. Since the maximum concentration of the silver salt solutions is less than 0.01 *M*, no correction for the selective sorption of water by the cotton need be made. Analysis shows that both of the curves of figure 1 conform approximately to the mass-action law, as would be expected.

* The effect of this phenomenon and the method of correcting for it are discussed elsewhere.[2]

Table 1 gives the results of measurements of the silver-binding capacity of the different samples of cotton which were immersed in 0.01 *M* solutions of silver nitrate, silver acetate, and silver *o*-nitrophenolate. Each of the results represents the mean of two determinations which agreed within 0.001 M-eq/g.

In accord with the above discussion, it was found that the silver-binding capacities of electrodialyzed dewaxed and electrodialyzed depectinized cotton increased as the hydrogen ion concentration of the solution in equilibrium with the samples decreased. When silver *o*-nitrophenolate was used, the final pH was above 7 and accordingly, the competition offered by the hydrogen ions is practically negligible. Thus, the values of 0.066 and 0.011 M-eq/g. obtained by titrating electrodialyzed dewaxed and electrodialyzed depectinized cottons, respectively, with this salt may be considered to be the base-binding capacities of these materials. The values obtained with this salt are in excellent agreement with the earlier estimates of the content of acidic groups.[2]

The data also show that when the cotton samples are not freed of their cationic ash by electrodialysis, the maximum silver-binding capacity is not attained under the conditions of these experiments, even though the pH of the equilibrium solution may be above 7. This may be explained as resulting from competition between silver ions and cations already on the

TABLE 1—SILVER-BINDING CAPACITY OF SAMPLES SOAKED IN 0.01 *M* SOLUTIONS OF VARIOUS SILVER SALTS

Sample	Solution	Silver bound	Equilibrium pH
		M-eq/g.	
Electrodialyzed dewaxed cotton	silver nitrate	0.021	3.22
Do.	silver acetate	.047	5.14
Do.	silver *o*-nitrophenolate	.066	7.12
Dewaxed cotton	silver nitrate	.036	5.12
Do.	silver acetate	.035	6.26
Do.	silver *o*-nitrophenolate	.046	7.60
Electrodialyzed depectinized cotton	silver *o*-nitrophenolate	.011	7.64
Lime-washed depectinized cotton	silver *o*-nitrophenolate	.009	7.64

fiber as shown in eq. 2. It should be noted that according to this equation silver ions of the solution are exchanged with cations such as calcium, rather than with hydrogen ions. The result of this exchange should be a smaller decrease of the initial pH values of the solutions. The data of table 1 show that the pH values of corresponding solutions are indeed higher for the samples that were not electrodialyzed, and as a result, competition of hydrogen ions for the acidic groups is lowered. Thus, when silver nitrate solutions are used, the hydrogen ion concentration of the solution in equi-

librium with the dewaxed cotton is considerably lower than that of a similar solution in equilibrium with the electrodialyzed dewaxed cotton, and a corresponding difference in silver-binding capacities is observed.

2. Combination with Calcium Ions

It has been shown elsewhere [4] that an estimate can be made of the number of acidic groups of cotton by engaging these groups with tightly held cations such as calcium, followed by electrodialytic determination of the cation content of the sample. In the present investigation, samples of electrodialyzed dewaxed and electrodialyzed depectinized cotton were treated for 2 hours with a large excess of a 0.01 *M* solution of calcium acetate and then washed in running distilled water for an additional 2 hours in order to remove unreacted calcium acetate. Determination of the cation contents showed that the electrodialyzed dewaxed sample bound 0.064 and the electrodialyzed depectinized, 0.010 M-eq/g. of calcium ion. Both values agree well with those obtained by titration with silver *o*-nitrophenolate as well as with the earlier estimates from the acid-binding determinations.[2]

3. The Acidic Groups in Depectinized Cotton

Since the same number of equivalents of calcium, silver, and hydrogen ions are bound by depectinized cotton, it may be concluded that both valences of the calcium are satisfied by the acidic groups of the fiber. This is of considerable significance inasmuch as it strongly indicates that the phenomenon being investigated is an acid-base equilibrium and not some indefinite adsorption process. Further evidence favoring this view has resulted from another type of experiment.

When electrodialyzed depectinized fibers * were treated for a short time with a solution of diazomethane in ether and then washed with a solution of calcium acetate, it was found that they bound less than 0.001 M-eq/g. of calcium. In other words, the acidic groups appear to be readily esterified by treatment with diazomethane, and are no longer available for reaction with base.

It was suggested earlier [2] that the acidic groups in depectinized cotton might well be a part of the cellulose molecule, especially since the experimental evidence did not favor the alternative possibilities that the groups might be present as the result of incomplete removal of the pectic substance or of the protein fraction during purification treatments. Assuming this to be true, the question still arises as to whether these groups were in the original cellulose or whether they were produced during the purification treatment of the fiber with alkali. In favor of the former possibility are the following experimental results. Dewaxed cotton contains 0.065 M-eq/g. of acidic groups. The pectic substance of the cotton contributes 0.055 M-eq/g. as shown by direct uronic acid determinations.[5] The difference, 0.010 M-eq/g., is exactly equal to the number of acidic groups found in the depectinized cotton.

It is conceivable, of course, that the acidic groups, other than those of the pectic substance, are in substances which are removed during purification of the fiber, and that the acidic groups found were formed by oxidation

* It is necessary to use fibers that are essentially ash-free, since the presence of cations on the acidic groups prevents their reaction with diazomethane.

of the cellulose during treatment with alkali.* This would necessitate the formation of 0.010 M-eq/g. of acidic groups, the same number as would be removed during the purification treatment. Such a coincidence appears to be rather unlikely. However, if it is assumed that such a reaction does take place and proceeds slowly, then it would be expected that the content of acidic groups would steadily increase during the treatment of the fiber with alkali. Such changes would be detectable by methods described in the present paper. If the reaction proceeds extremely rapidly so that it might go to completion even before complete removal of the pectic substance, the question whether the acidic groups in depectinized fibers are in the naturally occurring fiber or are produced during purification treatments cannot be answered at this time. Nevertheless, it appeared advisable to measure the base-binding capacity of samples purified for different lengths of time by extraction with a boiling 1-per cent solution of sodium hydroxide. The results are shown in table 2, and clearly indicate that no significant progressive changes in the acidic properties of the fiber have been produced, even after 16 hours of treatment with alkali. Thus, the possibility of slow formation of these acidic groups during the depectinizing treatments is also eliminated.

TABLE 2—CATION-BINDING CAPACITY OF COTTON SAMPLES AS A FUNCTION OF DURATION OF DEPECTINIZING TREATMENT

Duration of alkali treatment hr	Silver bound M-eq/g.	Calcium bound M-eq/g.
2	0.010	0.011
4	.010	.009
8	.011	.009
16	.011	.010

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* While precautions are taken to keep oxygen from the system, it is not possible under these conditions to exclude it rigorously.

Direct Processing of Whole Cotton

By GEORGE C. HOLROYD and FRANK K. CAMERON

ABSTRACT

Cellulose nitrates can be prepared from whole cotton without intermediate pulping. Cellulose acetates can be prepared from whole cotton but more readily from a high alpha-cellulose pulp. For cellulose ethers it appears necessary to prepare first a pulp.

IN early attempts to prepare a high-alpha cellulose from whole cotton, the woody components (stems, cusps, and seed cortex) required such long contact with the pulping reagents that some degradation of lint and loss of cellulose ensued. Murdock [11] has reported such an experience. This difficulty was met by pulverizing with a hammer mill. High-alpha cellulose pulps and in satisfactory yields have been obtained in the laboratory with standard procedures, viz., with alkali, sulfate, sulfite, and nitric acid. Milne and Cameron,[10] using chlorine as a pulping agent, obtained results comparing favorably with those obtained by Pomilio [13] from wheat straw; however, neither recovery nor grade was as satisfactory as when the previously cited methods were used.

Some of the non-cellulose components of whole cotton are removed in recovering the oil. The residue may be pulped easily. These facts suggest the possibility of making cellulose derivatives directly. It was recognized that different responses to reagents of the lint on the one hand and of the woody components on the other, might be more important than in pulping; and that the non-cellulose carbohydrates, not eliminated, might affect adversely the physical properties of the product. No experiments of this nature have been previously reported, although Hibbert [7] has privately described attempts to acetylate unpulped wood. The present work involves the preparation of cellulose nitrate, acetate, and ethyl ether.

CELLULOSE NITRATE

The raw material was the whole cotton plant, ground in a hammer mill to pass a 3/32″ screen, and extracted with petroleum ether. A nitrating mixture containing 64 per cent H_2SO_4, 21 per cent HNO_3, and 15 per cent H_2O was employed, fifty grams being used to each gram of oven-dry whole cotton. Nitration was conducted at room temperature, approximately 25° C. Under these conditions the stems and cusps required a longer time for nitration than the lint; it was found, however, that 24-hour treatment gave a product completely soluble in acetone or ethyl acetate.

To shorten the time required for nitration, preliminary treatment was attempted to render the cusps and stems more reactive. After treatment with dilute sodium hydroxide, nitration occurred readily. The material was

heated for four hours on the steam bath with ten times its weight of solution, two and five per cent solutions being used. After washing and dehydrating, both products were nitrated for one hour at 25° C. Complete solubility was attained in each case. When whole cotton passing a 5/16″ screen was treated by this procedure, the larger pieces of stems were not nitrated throughout. The nitrates were partially soluble in alcohol, indicating a nitrogen content within the range suitable for plastics. To secure a higher degree of nitration, a mixture containing 66 per cent H_2SO_4, 22 per cent HNO_3, and 12 per cent H_2O was used; the products were practically insoluble in alcohol.

The crude orange-colored nitrates were bleached by several modifications of the hypochlorite process. Direct treatment with hypochlorite yielded a light-green product, which was further bleached by two-hour refluxing with alcohol. When the material was first boiled with alcohol, less chlorine was required, but the alcohol treatment required several hours. One-hour refluxing with 50 per cent acetic acid, recommended by Hess [6] for stabilization, was still more effective in reducing the chlorine consumption. After subsequent hypochlorite treatment, the nearly-white material was completely bleached by a two-hour alcohol boil or by precipitation from acetone.

Films of the nitrates were flexible, clear, and tough. With nitrates intended for use in plastics or films, stability and viscosity determinations are of doubtful value.[3, 15] However, the nitrogen content produced by a particular method of preparation is important. Samples of untreated and alkali-treated whole cotton corresponding to ten grams of oven-dry material were nitrated, respectively, for 24 hours and one hour at 25° C. with the concentrated acid mixture containing 66 per cent H_2SO_4, 22 per cent HNO_3 and 12 per cent H_2O. The crude products were washed with water, refluxed for one hour with 50 per cent acetic acid, bleached with hypochlorite, and air-dried. Uniformity of the samples was insured by reprecipitation from acetone; the very small quantity of dirt remaining was removed by centrifuging the acetone solution. The following data were obtained:

	Conc. of Alkali in Pretreatment		
	0	2%	5%
Yield, oven-dry basis, grams	8.7	8.5	8.0
Chlorine consumed, grams	0.22	0.21	0.17
Nitrogen, per cent	12.2	11.1	11.4

The efficacy of nitric acid as a pulping agent indicated that it might be useful for this purpose also. Treatment with concentrated acid was therefore tried, at 2° and 25° C. After the mixture had stood for different periods, cold sulfuric acid of the calculated concentration was added at such a rate that the temperature did not rise above 25° C., and nitration continued for one hour at this temperature. With pretreatment as long as five hours at 2° C. and two hours at 25° C., the woody portions were not rendered completely soluble. Hot dilute acid was likewise ineffective. Two and five per cent aqueous solutions were used, with heating on the steam bath for one, two and four hours. With the five per cent solution in one hour, and with the two per cent solution in four hours, the lint was noticeably attacked. After washing with water and removing the water by steep-

ing in glacial acetic acid, the material, damp with acetic acid, was nitrated for one hour at 25° C. In no case was nitration complete.

CELLULOSE ACETATE

It is generally thought that cotton linters are the only suitable raw material from which to make cellulose acetate. Olsen,[12] however, has found that sulfite pulp, from many woods, after bleaching and mercerization, gives a uniform acetate with excellent physical properties. Consequently, a high alpha-cellulose pulp was prepared from whole cotton using the alcoholic nitric acid method of Aronovsky and Lynch [1] since it does not require pressure. First the stems were softened by an hour boil in 2 per cent caustic solution. The bleached and mercerized pulp gave an acetate which yielded clear, flexible films containing 0.04 per cent ash.

Whole cotton, steeped in 5% NaOH solution for 24 hours on the steam bath, then ground in water and steeped in a 17½% NaOH solution for 2 hours gave a product from which practically all the cellulose could be acetylated. The product, free from pentosans, formed acceptable films. But the long grinding, about 10 hours, and the difficult removal of ligneous material by long centrifuging, since filtration was unsuccessful, militate against the procedure. The crude acetate was readily bleached with permanganate.

Previous attempts by Milne [9] to prepare cellulose acetate from unpulped whole cotton were unsuccessful, acetylation of the woody portions without degradation of the cellulose being incomplete. Similar results were obtained in this investigation. Treatment with ten times its weight of a mixture of equal parts acetic acid and anhydrate was tried, with varying proportions of sulfuric acid and at different temperatures. With no preliminary treatment, a high concentration of sulfuric acid was required for esterification of the lint, the woody portions even then being only slightly attacked in 36 hours at 25° C. Introduction of gaseous chlorine into the mixture during acetylation had little effect on the yield.

Other possible catalysts for the reaction tried were sulfur dioxide and chlorine, according to the method of Barnett,[2] perchloric acid,[6] sulfuryl chloride, dry hydrogen chloride, and a mixture of hydrogen chloride and zinc chloride. Of these, only the last was appreciably effective. The mixed catalyst gave results comparable to those obtained with sulfuric acid.

In an effort to soften the woody components, the material was heated with 1½, 2 and 5 per cent sodium hydroxide for periods up to 24 hours. Disintegration of the stems on acetylation was incomplete in all cases. Finer grinding of the raw material and grinding in a mortar during acetylation, separately and in conjunction with alkali treatments, were also ineffective.

Treatment with mercerizing solution at 20° C. for periods as long as 24 hours also failed to result in complete acetylation, and the acetates still contained pentosan derivatives. Grinding in a ball mill with mercerizing solution resulted in destruction of the lint before the stems were disintegrated.

ETHYLCELLULOSE

Of the many modifications of the conventional ether synthesis which have been proposed for the manufacture of cellulose ethers,[14] a one-stage method of Dreyfus [4] appeared most suitable for this study. In this process the water content of the reaction mixture is reduced by the addition of de-

hydrating agents, with a consequent reduction in the quantities of alkali and etherifying agent required.

Ten grams of whole cotton was steeped in 50 grams of 50 per cent sodium hydroxide solution for two hours and pressed to remove excess caustic. Four hundred cc. of benzene was then added, the mixture cooled in ice, and 50 grams of calcium oxide gradually kneaded into the mass. Twenty-five grams of ethyl sulfate was added and the mixture refluxed for four hours, without apparent action on the cellulose.

As this experiment with untreated whole cotton was not successful, preliminary treatment with five per cent sodium hydroxide was given, with four-hour heating on the steam bath. For the formation of alkali-cellulose, varied concentrations of caustic solution were used. Samples of partially delignified whole cotton were treated with 40 per cent, 50 per cent, and saturated solutions of sodium hydroxide at room temperature for 2, 4, 24, and 48 hours. The mixture was pressed as nearly dry as possible and calcium oxide added as before. The mixture was refluxed with a large excess of ethyl sulfate for periods of 4, 15, and 24 hours, after which it was filtered and the benzene evaporated. A small residue of benzene-soluble material was obtained from samples given 24- and 48-hour alkali treatments and ethylated for 15 and 24 hours. The undissolved portion was washed with water, and the calcium hydroxide dissolved with hydrochloric acid. In all cases the residue contained insoluble woody material.

To avoid the necessity of using large quantities of acid, other methods of reducing the water content were tried. Alcohol, saturated with sodium hydroxide, and acetone were used to extract the water, as well as distillation with benzene. In all cases a residue of stems, apparently only slightly affected, remained after 24-hour ethylation.

When the pretreated material was heated under pressure for ten hours at 110–120° C. with excess sodium and ethyl bromide, there was no apparent action on the stems. Three such treatments resulted in etherification of all the plant components, but the cellulose was degraded to such an extent that films could not be obtained from the product.

Treatment with the etherifying agent prior to the addition of sodium hydroxide, as recommended by Miles,[8] was tried, both with whole cotton and with purified lint. The cellulose was heated overnight at 45° C. with ethyl sulfate containing a small quantity of sodium carbonate, cooled, mixed with 50 per cent sodium hydroxide and powdered caustic, and heated for ten hours at 45° C. Some decomposition of the ethyl sulfate occurred during the preliminary heating. In attempts to avoid this, the treatment was repeated with the addition of benzene as a diluent, but there was still evidence of decomposition of the ethyl sulfate, and the cellulose was not appreciably etherified. Ethyl bromide likewise failed to react under these conditions. At 110° C., it destroyed the cellulose.

CONCLUSIONS

1. Cellulose nitrates can be made from unpulped whole cotton after the oil has been removed. Preliminary treatment with dilute alkali to soften cusps and stems is advantageous.
2. Cellulose acetate has been made from a high alpha cellulose obtained from whole cotton. Cellulose acetate can be obtained from unpulped whole cotton but less readily than from the pulp.

3. Attempts to obtain cellulose ethers directly from unpulped whole cotton have not succeeded.

ACKNOWLEDGMENT

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Abstracts

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I. Fibers (Synthetic and Natural) and Fiber Analysis

Lousiness in Silk. T. Hayashi. *Rayon Text. Mo.*, Nov. 1940, V. 21, P. 52–3. (C)

Making Casein Fiber. E. O. Whittier and S. P. Gould. *Ind. Eng. Chem.*, V. 32, P. 906–7 (1940); cf. *C. A.*, V. 34, Col. 631; *Ibid.*, V. 34, Col. 5666. (C)

Modern Methods of Treating Wool to Prevent Shrinking. P. Vernioux. *Tiba*, V. 18, P. 19–25, 51–5 (1940); abs. in *C. A.*, V. 34, Col. 6084.

Chlorinated wool (I) in general dyes more unevenly and in darker tones than untreated wool (II). Brominated wool has an absorption capacity intermediate between I and II. The Drisol method (cf. Hall, *C. A.*, V. 33, Col. 8413) employing sulfuryl chloride affords complete and uniform penetration of the wool by the reagent. (C)

A New Process for the Manufacture of Cotton-like Yarns by Stretch-spinning of Cuprammonium-cellulose Solutions. A. Lottermoser. *Zellwolle, Kunstseide, Seide*, V. 45, P. 36 (1940); cf. *C. A.*, V. 34, Col. 5280; *Ibid.*, V. 34, Col. 6065.

A discussion of the Ger. pat. 668,695 (*C. A.*, V. 33, Col. 5186) and addn. pat. 677,429 (*C. A.*, V. 34, Col. 624). (C)

THE PROBLEMS OF ACETATE RAYON (INDUSTRY). K. W. Zachrich. *Angew. Chem.*, V. 53, P. 144–8 (1940); abs. in *C. A.*, V. 34, Col. 6820.

A discussion of the tech. difficulties caused by or involved in the following general aspects of cellulose acetate fibers: phys. properties of the fibers; the small degree of swelling; the resistance to chem. action; the thermal plasticity; the resistance to light; dyeing and printing; improvement of the textile properties. 28 references. (C)

VARIATIONS OF SHAPE AND AREA OF CROSS-SECTION IN SINGLE SILK FILAMENTS AND THEIR EFFECT UPON EXTENSIBILITY. A. C. Goodings and L. H. Turl (Ontario Research Foundation, Canada). *J. T. I.*, Sept. 1940, V. 31, P. T207–T218.

It is of importance in experiments dealing with the elastic properties of filaments to know the area of cross-section and its variations. To obtain information on such variations in silk to be used in the examination of these properties, silk was selected on the basis of high grading for evenness as determined by inspection on the seriplane; the data obtained are of interest as indicating the magnitude of the variations to be encountered in single filaments of a cultivated silk of recognized high grade. Although variations in shape are large, the variations in actual area of cross-section rarely exceed 20% of the mean. The form of the load-extension curve for silk filaments lends itself to simple mathematical treatment of the effect of variations in cross-sectional area on the extensibility of these filaments. It is shown that this effect is small, and that experimental curves, reduced to a stress basis, approximate closely to curves for ideally uniform filaments.

Methods for the determination of the mean area of cross-section of filaments were studied with reference to their application to relatively short segments of silk filaments. The method of weighing is considered to be the simplest and most reliable, and details are given of a micro-balance suitable for this purpose. (C)

VISCOSE FILAMENTS: DOUBLE REFRACTION. P. H. Hermans and P. Platzek. *Z. physikal. Chem.*, 1939, V. A185, P. 260–268; *C. A.*, 1940, V. 34, Col. 2585; abs. in *J. T. I.*, Sept. 1940, V. 31, P. A504.

Filaments were spun from 4 and 8 per cent viscose, stretched and treated in various ways, dried, cut wedge-shaped and examined under a polarizing microscope in sodium light. The specific double refraction increased with increase in the degree of swelling to which the filament was subjected before drying. As a function of the degree of extension to which the filament was initially subjected, the optical anisotropy measured in the dry state behaved qualitatively like the anisotropy in the swollen condition. (C)

THE VISCOSE PROCESS AND OXYGEN. A. Lottermoser. *Zellwolle, Kunstseide, Seide*, V. 45, P. 91–2 (1940); abs. in *C. A.*, V. 34, Col. 6065.

Viscose solns. absorb O very actively; they ripen differently depending on the O content of the atm. Thus a thick soln. is obtained in the presence of N or H while an atm. of O produces a much thinner liquid. When an alkali cellulose which has been preripened in O is xanthated and O is passed through the xanthate soln. during the aging period with simultaneous vigorous agitation, then the viscosity of the soln. is decreased so much that it approaches that of the NaOH soln. used. This decrease in viscosity dur-

ing the preripening stage and the constancy of the viscosity during the afterripening stage show that the macromols. of the cellulose are reduced during the preripening and not during the afterripening. Practical applications of these discoveries in manufg. operations are described. (C)

II. Yarns and Fabrics

Air Conditioning for Nylon Hosiery. H. L. Kempaner. *Text. Wld.*, Sept. 1940, V. 90, P. 101–2.

Nylon is highly susceptible to temperature variations, especially high temperatures, and to obtain best results with Nylon lower humidities and temperatures are required than are necessary with silk and it is desirable to control both the temperature and humidity. How this was accomplished in a Southern full-fashioned hosiery mill is described. (C)

Blends on Cotton Equipment. H. A. Franklin. *Text. Wld.*, Sept. 1940, V. 90, P. 94–7.

The blending of spun rayons and natural fibers and the processing of these blends on cotton machinery are discussed. (C)

"Character" in Worsted Yarns. "Garforth." *Text. Mfr.*, August 1940, V. 66, P. 302.

Discusses some of the less definable but valuable qualities of yarn. (C)

Cotton Fibers: Effect of Length on Spinning Quality. R. P. Richardson. *Text. Digest (Quart. J. Text. Assoc. India)*, 1940, I, No. 1, P. 7–10; abs. in *J. T. I.*, August 1940, V. 31, P. A428.

Some comments are offered on the influence of fiber length (and fineness) on the behavior of cotton in the various roller drafting zones. It is argued that the benefit of long staple as a contribution to yarn strength is due to the larger number of fibers per cross-section (giving greater surface of contact) and the better array of the fibers in drafting. (C)

Damage Appearing in Wool Cloth after Dyeing: The Development of Methods for Determining the Causes of. *Am. Dye. Rptr.*, 1940, V. 29, No. 22, P. P588–591, P595–598. (C)

Drying of Textiles. Henry Miedendorp, Jr. *Rayon Text. Mo.*, Nov. 1940, V. 21, P. 70. (C)

Durable Finishes on Rayon Fabrics. Philip Le Brun. *Text. Wld.*, Sept. 1940, V. 90, P. 98–9. (C)

The Effect of Pretreatment on the Specific Conductivity and Electrostatic Charge in Textile Fabrics. Bernhard Axhausen. *Klepzig's Textil.-Z.*, V. 43, P. 17–24, 201–6, 311–14, 324–5 (1940); *Chem. Zentr.*, 1940, I, 3601; abs. in *C. A.*, V. 34, Col. 6083.

During the spinning of artificial textile fibers, cotton and wool, there occur difficulties due to the formation of static electricity. The common means of prevention: (1) local withdrawal of current, (2) ionization of the atm., (3) high atm. humidity and (4) treatment of textiles with hygroscopic

or insulating oil or reviving agents are discussed critically. Significant phys. and technical measurements and properties are discussed as well as the effect of pretreatments of the textile on the production of these charges. (C)

ELECTRO-FIBER-COATED TEXTILE FABRICS. *Rayon Text. Mo.*, October 1940, V. 21, P. 56. (C)

III. CHEMICAL AND OTHER PROCESSING (NOT OTHERWISE CLASSIFIED)

RAYON AND STAPLE FIBRE: DYEING WITH VAT DYES. W. Hees. *Textilber.*, 1940, V. 21, P. 179–180; abs. in *J. T. I.*, July 1940, V. 31, P. A385; *C. A.*, V. 34, Col. 5665.

The uneven dyeing of regenerated cellulose rayon and staple fibre with Indanthrene dyes is discussed and the action of levelling agents is explained. It is pointed out that in hank dyeing processes the difficulty can to a large extent be overcome by the addition of a levelling agent, especially one having affinity for the dye, but improved methods are generally necessary for the machine dyeing of wound packages, such as bobbins and warp beams. Suitable modern methods, which include the pigment dyeing process, the stock vat dyeing process, the vat acid process, and a method in which dyeing starts at a temperature of 17–20° C., are briefly described. (C)

REGENERATED CELLULOSE FIBRES: DYEING PROPERTIES AND STRUCTURE. W. Schramek. *Leipz. Monats. Text. Ind.*, 1940, V. 55, P. 64–67; abs. in *J. T. I.*, June 1940, V. 31, P. A319; *C. A.*, V. 34, Col. 5284.

An account is given of investigations of the dyeing of regenerated cellulose fibres with basic and substantive dyes and colored diagrams are given showing cross-sections of viscose fibres at the beginning of dyeing operations with various dyes, when in equilibrium with the dye solution, and after washing with distilled water. Differences in dyeing properties between the different parts of a fibre are attributed to differences in structure. The evidence indicates that viscose fibre comprises an outer, very thin, skin in contact with a fibre mantle or shell, and a central core. When the viscose coagulates slowly the fibre mantle is characterized by a high degree of orientation and a high affinity for dyes, especially basic dyes. With increasing speed of coagulation of the viscose, the degree of orientation and the affinity for dyes of this mantle decrease. The mantle is very dense and dyes of large particle size cannot diffuse through it. The thickness of the mantle decreases with increasing ripening of the viscose. The core of a fibre obtained from a spinning solution in which the cellulose aggregates are to a great extent broken up takes up dyes quickly but does not retain them on washing. With less degraded cellulose the affinity for dyes increases. The affinity of the core for dyes also increases with increasing ripening of the viscose. Under certain conditions the core may show an outer zone of high affinity which must be attributed to a high degree of orientation, and this degree of orientation may in certain cases exceed that of the fibre mantle. The fibre core is, however, always less dense than the fibre mantle, and substantive dyes in a suitable state of division can diffuse through it. The highest density is, therefore, not always associated with

the highest degree of orientation. With increasing ripening of the viscose the zone of higher orientation extends further into the interior of the core, while the less orientated mantle decreases in thickness. (C)

SCUMMING OR "TARNISH" ON PRINTED GOODS AND ITS CAUSES. Francis H. M. New. *Rayon Text. Mo.*, August 1940, V. 21, P. 60.

The causes of "scumming" of "whites," and their prevention and remedies are discussed. (C)

IV. RESEARCH METHODS AND APPARATUS

COMPARATIVE STUDIES IN THE DETERMINATION OF THE QUALITY OF FIBERS IN THE FLAX STEM. H. Ulbricht. *Klepzig's Textil.-Z.*, 1939, V. 42, P. 447; *C. A.*, 1940, V. 34, Col. 1179; abs. in *J. T. I.*, August 1940, V. 31, P. A448.

The determination of the quality by various methods used in several laboratories showed good agreement of the results. By Tobler's micro method it is possible to examine very small amounts for quality of the textile; it can also be used to test large amounts if care is taken to obtain an average sample. (C)

DEPTH OF FOCUS OF MICROSCOPE OBJECTIVES. D. G. Beadle. *Nature*, June 29, 1940, V. 145, P. 1018–19; abs. in *Eastman Kodak Abs. Bull.*, October 1940, P. 537.

The variation in number of fine dust particles photographed at different settings of the microscope objectives indicates that the depth of focus of microscope objectives is less than the theoretical formulas indicate. (S)

DETERMINATION OF FIBER FINENESS OF A MERINO WOOL SAMPLE. V. Bosman and C. M. van Wyk. *Onderstepoort J.*, 1939, V. 13, P. 401–413; abs. in *J. T. I.*, Oct. 1940, V. 31, P. A568.

A description of the South African technique for preparing and mounting a wool sample and for measuring and recording the results. 250 measurements are generally sufficient to bring the permissible error to within 3% of the mean, a chart being given to show the relationships between the statistical constants and the number of fibers necessary for measurement, the values given being for a 5% probability level of significance. The question of the human element among observers is discussed. Difficulties are stated peculiar to the weight-length method for determining fiber fineness, which has been suggested for arbitration purposes. (C)

DETERMINATION OF WEARING QUALITIES BY AIR FLOW MEASUREMENTS. Erwin J. Saxl. *Am. Dye. Rptr.*, 1940, V. 30, No. 24, P. P637–P641. (C)

EFFECT OF HUMIDITY AND TEMPERATURE ON THE YOUNG'S MODULUS OF KERATIN FIBERS. H. J. Woods. *Proc. Leeds Phil. and Lit. Soc., Sci. Section*, 1940, V. 3, P. 577–583; abs. in *J. T. I.*, Oct. 1940, V. 31, P. A569.

Using high rates of extension within the Hooke's law region of the load/extension curve, a series of measurements of Young's modulus can be

made with a single fiber. For humidity changes the results agree with Speakman's statistical values; the water adsorbed at low humidities has less effect than the remainder in reducing Young's modulus. The temperature effect for all types of hair is small compared with the large temperature variation of relaxation phenomena. For dry fibers the temperature coefficient of Young's modulus between 0° and 100° C. is of the order -2.5×10^{-3} per degree, and a somewhat similar value is obtained for wet fibers between 40° and 90° C. Usually, particularly with human hair, Young's modulus for wet fibers decreases much more rapidly as the temperature rises to 40° C. than above this temperature. (C)

V. Pure Science, Economics and Misc.

A Suspended Photomicrographic Camera. W. S. Hastings. *J. Biol. Phot. Assoc.*, December 1939, V. 8, P. 55–9; abs. in *Eastman Kodak Abs. Bull.*, April 1940, P. 178.

A description and drawings are given of photomicrographic equipment which is suspended from the ceiling with a vertical optical axis, thus requiring an over-all floor space of only 19 inches by 27 inches and having the advantage of a horizontal stage. The long frame, which serves as an optical bed, consists of four steel bars whose overhead support floats on a slightly inflated rubber ring. A reflex camera is used. The illumination system at the bottom is on a horizontal bed, the beam being reflected vertically with a mirror. (S)

Textile Casein: Characteristics of. E. Pijanowski. *Poradnik Mleczarski i Fajczarski*, 1938, No. 23, P. 19–22 (in Polish with English summary); *Dairy Sci. Abs.*, V. 1, No. 1, P. 24; abs. in *C. A.*, 1940, V. 34, Col. 2609.

The general uses and properties of casein are given and a method for the prepn. of textile casein is outlined. Two samples of Italian manuf., 2 made by the Dutch method, 2 made locally and 1 sample of casein pptd. with lactic acid were fully analyzed chemically and physically and on this basis a standard is suggested for the compn. of textile casein. The Dutch method of manuf. dispenses with filters and is economical. (C)

Textile Fiber Atlas. Werner von Bergen and Walter Krauss. *Rayon Text. Mo.*, 1940, V. 21. Part I. Wool. January, P. 57. II. Specialty Hair Fibers. February, P. 47–50. III. Fur Fibers. March, P. 53–7. IV. Minor Hair Fibers. May, P. 53–7. V. Silks. June, P. 49–53. VI. Cotton and Minor Seed Hairs. July, P. 49–52. VII. Bast Fibers. August, P. 49–52. VIII. Rayon. October, P. 49–53. IX. Protein and Synthetic Fibers. Nov., P. 55–8. X. Structural Fibers. Dec., P. 47–50.

This series of articles, each of which is accompanied by plates containing accurate photomicrographs of the various parts of the fibers discussed, is to be published in book form upon completion. (C)

Textile Mill Waste Water: Purification. M. Kehren. *Zellwolle, Kunstseide, Seide*, 1939, V. 44, P. 405–8; abs. in *J. T. I.*, April 1940, P. A212.

Tests of the efficiency of precipitation treatments with (1) aluminum sulphate, (2) caustic lime, (3) hydrous ferrous sulphate and (4) hydrochloric acid for the purification of a sample of waste water from a dye works are

described, costs are compared, and the suitability of the processes for industrial application is discussed. An iron-carbonic acid precipitation treatment which is being used successfully on a large scale for the purification of waste water containing dyes is briefly described. In this process, after a settling treatment, the water is treated with carbon dioxide-containing flue gases, and cast iron chips are then added and air is passed through the mixture. Rayon and staple fibre producers have to dispose of very large quantities of waste water. Acid and alkaline wastes can be mixed and after settling, with or without the addition of cheap precipitating agents, run into large rivers. Patent processes for the recovery of chemicals from waste waters from cuprammonium and viscose rayon plants are mentioned and the importance of recovery of fatty acids from wool washing and fulling waters is pointed out. (C)

TEXTILES: PROPAGATION OF TEMPERATURE CHANGES IN HUMID ATMOSPHERES. G. King, A. B. D. Cassie and S. Baxter. *Trans. Faraday Soc.*, 1940, V. 36, P. 445–453, 453–458, 458–465; abs. in *J. T. I.*, May 1940, P. A266. (C)

THALLOUS ETHYLATE: METHOD FOR ESTIMATING THE INTERNAL SURFACE OF CELLULOSE BY MEANS OF. Chapin A. Harris and C. B. Purves. *Paper Trade J.*, Feb. 8, 1940, P. 63–7.

Thallous ethylate, in dilute ethereal or benzene solution, and dry, mercerized ramie gave a thallium cellulose in which the metal substituted hydroxyl hydrogen. The thallium cellulose when methylated gave a product with a methoxyl content of $1.45 \pm 0.3\%$. One hydroxyl in every forty had thus been substituted by thallium and the effective internal surface of the mercerized ramie was accordingly about 4×10^5 sq. cm. per gram. (C)

UNDESIRABLE ELECTROLYTIC PHENOMENA IN THE TEXTILE INDUSTRY. H. Rammensee. *Klepzig's Textil-Z.*, 1939, V. 42, P. 481–2; abs. in *C. A.*, 1940, V. 34, Col. 1184.

Several examples of unwanted electrolysis, corrosion, etc., in com. textile operations are discussed. (C)

USE OF STANDARDS IN THE TEXTILE INDUSTRY. William H. Whitcomb. *Canadian Text. J.*, June 7, 1940, V. 57, P. 28–34, 48–9, 53, 55.

The author, who is Secretary of Committee D-13 of the A. S. T. M., presented this paper before the Federation of Textile Technical Associations of Canada at their first annual meeting at Ottawa, June 1. He described how textile standards have been developed in this country, their benefit to the manufacturer, distributor and ultimate consumer; and emphasizing the fact that co-operation has been the keynote of standardization. (C)

VERTICAL ILLUMINATION FOR OPAQUE PHOTOMICROGRAPHY. J. G. Pratt. *Amer. Annual of Photography*, 1940, V. 54, P. 237–9; abs. in *Eastman Kodak Abs. Bull.*, April 1940, P. 179.

A concise microscope mirror with a one-half-inch aperture cut in the center is used. The mirror is placed in such a position that the lens shoots through the aperture to the specimen and can be adjusted to the best advantage while the image is viewed on the ground glass. Detailed directions for the photographing of several typical specimens are given. (S)